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Argonne National Laboratory

THE EBR-II MATERIALS-SURVEILLANCE PROGRAM: II. Results of SURV-2

by

Sherman Greenberg, Robert V. Strain, and Earl Ebersole

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Part I is ANL-7624

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ABSTRACT

Eight subassemblies containing 15 alloys used in the primary system of EBR-II, and shield graphite canned in Type 304 stainless steel, are being irradiated in the EBR-II core at 700°F for varying periods of time and neutron dosage. Half of the alloy specimens are exposed directly to reactor sodium, and the remainder are sealed in helium. The alloys include AMPCO Grade18 aluminum bronze; Stellite 6B; Inconel X-750; T-1 tool steel; Berylco-25; Types 304, 347, 416, and 420 stainless steel (the Type 304 in four variations); Type 17-4 PH stainless steel; and tantalum.

The second subassembly (SURV-2) has been removed and subjected to a limited examination after 1581 days in the reactor core and an estimated average fluence of 7×10^{19} n/cm^2 at energy levels >0.82 MeV (3×10^{21} n/cm^2 total). Of the materials exposed, only the Berylco-25 and tantalum experienced appreciable weight loss, and thus appear to be unsuitable for use in contact with sodium under the conditions of test. Type 304 stainless steel (from the shield-graphite cans) increased slightly in room-temperature yield strength but remained ductile. There was no evidence of loss of integrity of the canned-graphite neutron-shield structures. Tantalum samples experienced density decreases that were a function of fluence and equivalent to a maximum swelling of 0.4%.

I. INTRODUCTION

A long-term irradiation test program is in progress to monitor the behavior of the materials in service (primarily those in long-term service) in the primary-system sodium of EBR-II and of the neutron-shield graphite. The program involves the exposure and evaluation of 10 subassemblies containing the materials of interest in suitable form.

THE EBR-II MATERIALS-SURVEILLANCE PROGRAM:

II. Results of SURV-2

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ABSTRACT

Eight subassemblies containing 13 alloys used in the primary system of EBR-II, and shield graphite canned in Type 304 stainless steel, are being irradiated in the EBR-II core at 700°F for varying periods of time and neutron dosage. Half of the alloy specimens are exposed directly to reactor sodium, and the remainder are sealed in helium. The alloys include AMPCO Grade 18 aluminum bronze, Stellite 6B, Inconel X-750, H-11 tool steel, Berylco-25, Types 304, 347, 416, and 420 stainless steel (the Type 304 in four variations), Type 17-4 PH stainless steel, and tantalum.

The second subassembly (SURV-2) has been removed and subjected to a limited examination after 1581 days in the reactor core and an estimated average fluence of 1 x 10^{22} n/cm^2 at energy levels >0.82 MeV (3 x 10^{22} n/cm^2 total). Of the materials exposed, only the Berylco-25 and tantalum experienced appreciable weight loss, and these appear to be unsuitable for use in contact with sodium under the conditions of test. Type 304 stainless steel (from the shield-graphite cans) increased slightly in room-temperature yield strength but remained ductile. There was no evidence of loss of integrity of the canned-graphite neutron-shield structures. Tantalum samples experienced density decreases that were a function of fluence and equivalent to a maximum swelling of 0.4%.

I. INTRODUCTION

A long-term irradiation test program is in progress to monitor the behavior of the materials in service (primarily those in long-term service) in the primary-system sodium of EBR-II and of the neutron-shield graphite. The program involves the exposure and evaluation of 10 subassemblies containing the materials of interest in suitable form.

A detailed description of the program and experimental method, and the results of SURV-1, have been reported.* The present report gives the results of a limited examination of SURV-2. This report is intended for use in conjunction with ANL-7624, which gives a detailed description of sample materials, types, and arrangements and experimental procedures. In particular, the loading diagram for SURV-2 is identical with that for SURV-1 (Table V, ANL-7624, p. 20).

Because the total fluence received by SURV-2 was below that thought to be necessary to significantly alter the mechanical properties of stainless steel, the mechanical-property determinations made for SURV-1 were not repeated for SURV-2. The graphite-can interaction has been studied in greater detail than for SURV-1, since the continued successful operation of EBR-II depends on the integrity of canned-graphite shield structures. The mechanical-property samples from SURV-2 have been stored so that measurements can be made in the future if desirable. In addition, high-temperature tensile properties of the Type 304 stainless steel tensile samples will be determined at Battelle Northwest Laboratories and reported separately.

II. DOSIMETRY AND EXPOSURE

SURV-2 was removed from the reactor on June 28, 1969. It had been in the reactor in position 12E1 for 1581 days at a temperature of approximately 700°F and had received a total exposure of 26,274 MWd. The estimated average total fluence, as calculated from one-dimensional, 22-group diffusion theory, was 3×10^{21} n/cm^2.

Wire flux monitors, in reactor position 12E1, were used to determine the relative fast fluence as a function of position in the subassembly. The upper ends of the wires were 10.58 in. above the core centerline.

The neutron reaction for each of the four wires is:

Copper:	$^{63}Cu(n,\alpha)^{60}Co$
Iron:	$^{54}Fe(n,p)^{54}Mn$
Nickel:	$^{58}Ni(n,p)^{58}Co$
Titanium:	$^{46}Ti(n,p)^{46}Sc$

Each wire was cut into 1-in. long sections. Each section was weighed, and then analyzed by gamma spectrometry (January 22, 1970). The activity in counts/min, summed under the photopeak and corrected for sample weight, was converted to dis/sec per gram of wire. The data are summarized in Figs. 1-4. The curves in the figures are useful in assessing the relative exposure of the samples to fast neutrons as a function of position in the subassembly.

*The EBR-II Materials-surveillance Program: I. Program and Results of SURV-1, ANL-7624 (Sept 1969).

A detailed description of the program and experimental method, and the results of SURV-1, have been reported.* The present report gives the results of a limited examination of SURV-2. This report is intended for use in conjunction with ANL-7624, which gives a detailed description of sample materials, types, and arrangements and experimental procedures. In particular, the loading diagram for SURV-2 is identical with that for SURV-1 (Table V, ANL-7624, p. 20).

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II. DOSIMETRY AND EXPOSURE

SURV-2 was removed from the reactor on June 25, 1969. It had been in the reactor in position 12E1 for [illegible] days at a temperature of approximately 700°F and had received a total exposure of 26,274 MWd. The estimated average total fluence, as calculated from one-dimensional, 22-group diffusion theory, was 3×10^{21} n/cm^2.

Wire flux monitors, in reactor position 12E1, were used to determine the relative fast fluence as a function of position in the subassembly. The upper ends of the wires were 10.56 in. above the core centerline.

The neutron reaction for each of the flux wires is:

Copper	$^{63}Cu(n,\alpha)^{60}Co$
Iron	$^{54}Fe(n,p)^{54}Mn$
Nickel	$^{58}Ni(n,p)^{58}Co$
Titanium	$^{46}Ti(n,p)^{46}Sc$

Each wire was cut into 1-in.-long sections. Each section was weighed, and then analyzed by gamma spectrometry (January 22, 1970). The activity in counts/min, summed under the photopeak and corrected for sample weight, was converted to dis/sec per gram of wire. The data are summarized in Figs. 1-4. The curves in the figures are useful in assessing the relative exposure of the samples to fast neutrons as a function of position in the subassembly.

*The EBR-II Materials-surveillance Program: I. Program and Results of SURV-1, ANL-7624 (Sept 1969).

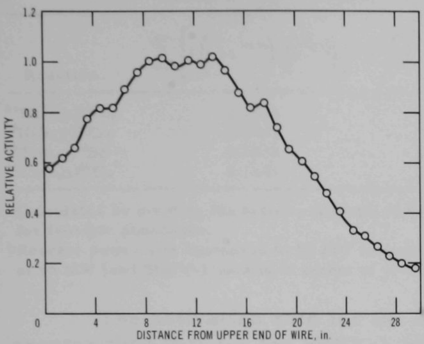

Fig. 1. ^{60}Co Activity of Copper Wire That Monitored SURV-2 Flux

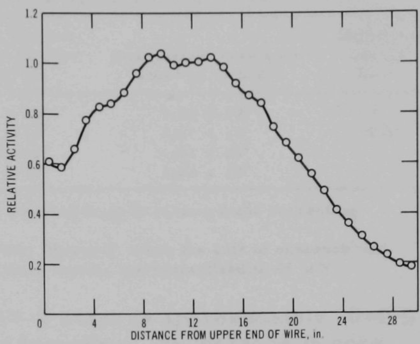

Fig. 2. ^{54}Mn Activity of Iron Wire That Monitored SURV-2 Flux

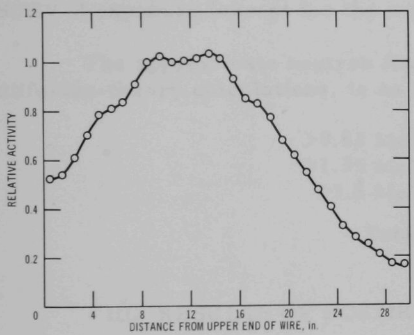

Fig. 3. ^{58}Co Activity of Nickel Wire That Monitored SURV-2 Flux

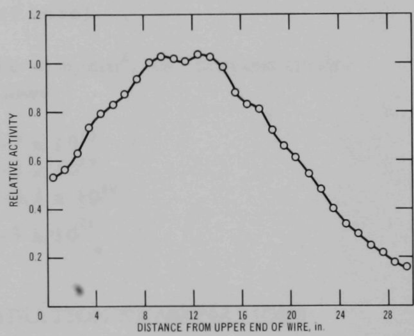

Fig. 4. ^{46}Sc Activity of Titanium Wire That Monitored SURV-2 Flux

The equilibrium activation rate at 45 MW has been calculated from the data for wire activity for a position ~1 in. above the core centerline, where the relative activity is taken as unity. The calculations are summarized in Table I. To correct for the intermittent operation of the reactor, the quantity

$$f\left(1 - e^{-\lambda t_1}\right)\left(e^{-\lambda t_2}\right)$$

was evaluated for each reactor run, where f = fraction of 45 MW exposure, t_1 = irradiation time, and t_2 = decay time from end of irradiation to counting time. The values of

$$f\left(1 - e^{-\lambda t_1}\right)\left(e^{-\lambda t_2}\right)$$

[illegible]re summed.

TABLE I. SURV-2 Neutron Activation Calculated from Flux Wires

Reaction	$\sum\left[f\left(1-e^{-\lambda t_1}\right)\left(e^{-\lambda t_2}\right)\right]$	Activity, dis/sec per gram of Wire (1/22/70)	Equilibrium Activation Rate[a] at 45 MW[b]	Threshold Energy, MeV
$^{54}Fe(n,p)^{54}Mn$	0.2595	8.12×10^6	5.38×10^8	~3
$^{58}Ni(n,p)^{58}Co$	0.0767	3.97×10^7	7.61×10^8	~2.9
$^{46}Ti(n,p)^{46}Sc$	0.1008	8.22×10^5	1.03×10^8	-
$^{63}Cu(n,\alpha)^{60}Co$	0.1475	2.67×10^5	2.62×10^6	-

[a]Calculated by dividing the activity (column 3) by the summed term in column 2 and correcting for isotopic abundance.

[b]Reactor power was increased to 50 MW in August 1968. However, since the bulk of exposure was at 45 MW (and SURV-1 data is in terms of 45 MW), these results are normalized to 45 MW.

The activation rate for the SURV-2 wires is approximately 30-40% greater than that for the SURV-1 wires because of the increase in core size (to accommodate an increased number of experiments) during the latter part of the irradiation period. Because the half-lives of the isotopes other than ^{60}Co are relatively short compared with the exposure time, the activity measured was that produced during the latter portion of the SURV-2 exposure (except for the copper wire).

The approximate neutron fluence in n/cm^2, as obtained from diffusion-theory calculations, is as follows:

$$>0.82\ \text{MeV}\text{--}7 \times 10^{19}$$
$$>1.35\ \text{MeV}\text{--}2 \times 10^{19}$$
$$>3.8\ \text{MeV}\text{--}0.3 \times 10^{19}$$
$$\text{Total}\text{--}3 \times 10^{21}$$

III. RESULTS OF POSTIRRADIATION EXAMINATIONS

A. Weight-change Data

The weight-change data (obtained by using the hardness-sample cylinders) are summarized in Table II. As was true for SURV-1, only the Berylco-25 and tantalum samples showed consistently high weight losses. (There are no longer any Berylco-25 components operating in the reactor.) One sample each of the Ampco 18 aluminum bronze and T-1 tool steel showed unusually high weight losses. These weight losses are isolated cases, and components of these materials continue to perform satisfactorily in EBR-II. Therefore, it is believed that these unusual results (which will be subject to confirmation by examination of SURV-3) are due to experimental error and are not cause for concern.

TABLE II. Weight Changes of Hardness Samples from SURV-2

Material	Sample[a] No.	Preirradiation Weight, g	Weight Change,[b] mg
Aluminum Bronze--Ampco Grade 18	A-1	16.1235	-1.1
	A-2	16.0560	-1.5
	A-3	16.0648	-9.7
	A-4	16.0629	-1.0
	A-5	16.0239	-0.0
Stellite 6B	B-1	18.0991	+0.3
	B-2	18.0889	+0.3
	B-3	18.0865	+0.1
	B-4	18.1008	+0.6
	B-5	18.1005	+0.7
Inconel X-750 (Heat-treated to hardness of Rockwell C 37-40)	C-1	17.7736	-1.1
	C-2	17.7620	-1.0
	C-3	17.7684	-1.1
	C-4	17.6870	-1.0
	C-5	17.7699	-1.2
Type 420 Stainless Steel (Heat-treated to hardness of Rockwell C 40-45)	D-1	16.3578	-0.2
	D-2	16.3498	-0.9
	D-3	16.3557	-0.5
	D-4	16.4963	-0.9
	D-5	16.5042	-0.5
Tool Steel--T-1 (Heat-treated to hardness of Rockwell C 55-60)	E-1	18.6689	+0.2
	E-2	18.6770	-0.5
	E-3	18.6598	+0.1
	E-4	18.6753	-9.3
	E-5	18.7068	+1.1
Type 347 Stainless Steel	F-1	16.9494	-1.0
	F-2	16.9453	-0.4
	F-3	16.9346	-0.5
	F-4	16.9265	-0.6
	F-5	16.9272	-0.6
Type 416 Stainless Steel (Heat-treated to hardness of Rockwell C 30-34)	G-1	16.3470	0.0
	G-2	16.3083	+0.2
	G-3	16.3447	-0.3
	G-4	16.3548	-1.1
	G-5	16.3550	-0.7
Beryllium Copper--Berylco-25 (Heat-treated to hardness of Rockwell C 41-45)	H-1	17.9677	-137.7
	H-2	17.9745	-333.9
	H-3	17.9862	-491.6
	H-4	17.9594	-3.6
	H-5	17.9843	-1.1
Type 304 Stainless Steel with Boron	I-1	16.7669	-1.5
	I-2	16.7736	-1.1
	I-3	16.8037	-1.8
	I-4	16.5024	-0.7
	I-5	16.8292	-0.2
Type 17-4 PH Stainless Steel (Heat-treated to hardness of Rockwell C 36-41)	J-1	16.6606	-0.8
	J-2	16.6748	-0.5
	J-3	16.6930	-1.0
	J-4	16.6408	-0.5
	J-5	16.7058	-0.6
Type 304 Stainless Steel	K-1	16.8490	-0.3
	K-2	16.8591	-1.8
	K-3	16.8633	-0.3
	K-4	16.8248	-0.3
	K-5	16.8222	-0.4
Tantalum	M-1	36.1254	-9.7
	M-2	36.1815	-29.9
	M-3	36.1617	-11.5
	M-4	36.1365	-18.4
	M-5	36.1470	+0.5

[a]For each material listed, the first four samples were exposed directly to the sodium of the reactor primary system. The fifth sample was sealed in a capsule containing helium.
[b]Exposed surface area was 7.8 cm^3.

B. Density-change Data

For all materials except Stellite 6B, Inconel X-750, T-1 tool steel, and tantalum, the postexposure densities were within the range measured before exposure.

Stellite 6B and Inconel X-750 increased in density by 13 and 17 mg/cm^3, respectively. The initial densities were $8.376\ ^{+0.003}_{-0.011}$ and $8.267\ ^{+0.007}_{-0.013}\ g/cm^3$, respectively.

The tool-steel samples decreased in density by from 14 to 18 mg/cm^3. Initial density was $8.656 \pm 0.003\ g/cm^3$. Since there was no consistent relationship between density decrease and sample position (i.e., fluence), it is speculated that the change is the result of thermal conditions rather than neutron fluence.

The decrease in tantalum density (54-77 mg/cm^3) was the greatest of any of the samples and varied directly with fluence. The maximum decrease in density was equivalent to a swelling of approximately 0.4%. The tantalum data (summarized in Table III) are of particular interest because of the relatively large change apparently caused by neutron exposure.

TABLE III. Density Decrease[a] of Tantalum Hardness Samples from SURV-2

Sample No.	Environment	Position, in. above Bottom of Core	Density Decrease, mg/cm^3
M-1	Sodium	1.00	61
M-5	Helium	1.00	54
M-2	Sodium	6.75	69
M-6	Helium	6.75	69
M-3	Sodium	15.50	63
M-7	Helium	15.50	55
M-4	Sodium	4.75	69
M-8	Helium	4.75	77

[a]The average preexposure density was $16.685\ ^{+0.005}_{-0.012}\ g/cm^3$.

There was essentially no difference between samples exposed to sodium and those exposed to helium.

C. Hardness-change Data

At the time of the preparation of the SURV-1 report (ANL-7624), hardness data for the samples exposed in helium were not available. The

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For all materials except Stellite 6B, Inconel X-750, T-1 tool steel, and tantalum, the postexposure densities were within the range measured before exposure.

Stellite 6B and Inconel X-750 decreased in density by 13 and 17 mg/cm^3, respectively. The initial densities were $8.376^{+0.007}_{-0.011}$ and 8.267 ± 0.007 g/cm^3, respectively.

The tool-steel samples decreased in density by from 14 to 18 mg/cm^3. Initial density was 8.650 ± 0.003 g/cm^3. Since there was no consistent relationship between density decrease and sample position (i.e., fluence), it is speculated that the change is the result of thermal conditions rather than neutron fluence.

The decrease in tantalum density (54-77 mg/cm^3) was the greatest of any of the samples and varied directly with fluence. The maximum decrease in density was equivalent to a swelling of approximately 0.4%. The tantalum data (summarized in Table III) are of particular interest because of the relatively large change apparently caused by neutron exposure.

TABLE III. Density Decreases[a] of Tantalum Hardness Samples from SHRV-2

Sample No.	Environment	Position, in. above Bottom of Core	Density Decrease, mg/cm^3
M-1	Sodium	1.00	61
M-5	Helium	1.00	54
M-2	Sodium	6.75	69
M-6	Helium	6.75	64
M-3	Sodium	15.50	63
M-7	Helium	15.50	65
M-4	Sodium	4.75	69
M-8	Helium	4.75	77

[a]The average preexposure density was $16.685^{+0.005}_{-0.006}$ g/cm^3.

There was essentially no difference between samples exposed to sodium and those exposed to helium.

C. Hardness-change Data

At the time of the preparation of the SHRV-1 report (ANL-7024), hardness data for the samples exposed in helium were not available. The

Berylco-25 exposed to sodium had softened appreciably--a decrease in the average Vickers Hardness Number (500-g load) from 320 to the range of 161-176. In the absence of the helium-exposure data, it was speculated that the sodium environment contributed to the softening.

Since then, data from the helium-exposed samples from SURV-1 have become available. The average Vickers Hardness Number (500-g load) is 160. Thus, the sodium, although it severely corroded the Berylco-25, was not responsible for the softening. As stated in ANL-7624, the microstructure indicated that the Berylco-25 had overaged as the result of the SURV-1 exposure, and the overaging is responsible for the softening. A sodium-exposed Berylco-25 sample from SURV-2 had an average Vickers Hardness Number (500-g load) of 160; i.e., there is no significant change in hardness as a result of the increased exposure. It will be necessary to examine the samples exposed in the storage basket to distinguish between effects of thermal and neutron exposure.

D. Examination of Graphite and Cans

Measurement of external dimensions of the graphite cans showed no appreciable dimensional changes (to ±0.001 in.) or torsional distortion (to ±0.002 in.). When the cans were cut open, there was no evidence of sodium in-leakage to the graphite. In SURV-1, sodium leaked into the annulus between the cover plate and the end cap of one can. There was no such leakage of sodium in SURV-2.

1. Graphite

Table IV lists the density of the graphite after the SURV-2 exposure and the density of unirradiated material. The density of the unirradiated material was determined by weighing accurately measured machined blocks. This method was also used for SURV-1. However, it could not be used for SURV-2 because of mechanical difficulties, and the density was determined using a method based on ASTM Procedure C97-47 (Reapproved 1958) for natural building stone. Unirradiated canned graphite blocks have become available. Density of this graphite will be determined by using the ASTM procedure. Results will appear in the SURV-3 report.

TABLE IV. Effect of Exposure on Density of Graphite

Type of Graphite	Density, g/cm^3	
	Before Exposure	After Exposure
Plain	1.5419-1.6376[a]	1.6933
Borated	1.5601	1.5639

[a]Range for six lots.

Although the methods of measurement are different, it is likely that the increase in density is real and that there has been no swelling. An increase in density at exposures up to ~1.5 x 10^{22} nvt has been reported for somewhat comparable conditions in the Dounreay Fast Reactor.*

2. Can Material

Tensile samples (from the unwelded faces)** were cut and tested at room temperature. The results, together with values for similar samples cut from an unexposed can, are shown in Table V.

TABLE V. Tensile Properties of SURV-2 Graphite-can Material

	Unirradiated Can Material		SURV-2 Irradiated Can Material	
Property[a]	Average	Range	Average	Range
Yield strength, psi	39,400	38,300-40,400	42,500	40,900-44,100
Ultimate strength, psi	93,500	92,500-94,700	90,200	87,900-92,400
Total elongation, %	44	42-45	41	37-44
Reduction in area, %	57	56-59	50	45-56

[a]Obtained at strain rate of 0.012 min^{-1}.

The SURV-2 exposure had only slight effect on room-temperature tensile properties. The data for reduction in area and elongation show that the material is still very ductile after exposure in SURV-2.

Sections of the graphite cans, through the face welds, were bent 180° over a 1/2-in.-dia mandrel. The weld joint was parallel to the axis of the mandrel and at the apex of the bend. There was no cracking or other unusual effect.

The thickness of the carburized zone on the interior of the cans was similar to that after the SURV-1 exposure---~0.03-0.04 mm. However, in contrast with SURV-1, in which all graphite was easily removable from the cans, some sticking of the graphite occurred in SURV-2. Optical metallographic and electron-microprobe examination revealed no evidence of interaction between the graphite and stainless steel can material or of any unusual attack on the sodium-exposed surface of the stainless steel.

IV. DISCUSSION AND CONCLUSIONS

Examination of the materials exposed in SURV-2 indicate no materials-related problems or potential problems in the continued operation of EBR-II.

*Reynolds, W. N., Physical Properties of Graphite, Elsevier Publ. Co., Amsterdam (1968), Ch. 7.

**The can is formed by welding two half (channel-shaped) sections.

Although the methods of measurement are [illegible], it is likely that the increase in density is real [illegible] graphite [illegible] swelling. An increase in density at exposures up to $1.9 \times 10^{[illegible]}$ nvt has been reported for somewhat comparable conditions in the Dounreay Fast Reactor.*

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Property*	Average	Range	Average	Range
Yield strength, psi	39,400	38,300-40,400	42,500	40,900-44,100
Ultimate strength, psi	93,500	92,500-94,700	90,200	87,900-92,400
Total elongation, %	44	42-45	41	37-44
Reduction in area, %	58	56-59	50	45-56

*Obtained at strain rate of 0.012 min^{-1}.

The SURV-2 exposure had only slight effect on room-temperature tensile properties. The data for reduction in area and elongation show that the material is still very ductile after exposure in SURV-2.

Sections of the graphite cans, through the face welds, were bent 180° over a 1/2-in.-dia mandrel. The weld joint was parallel to the axis of the mandrel and at the apex of the bend. There was no cracking or other unusual effect.

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**[illegible]

*Reynolds, W. N., [illegible]

*Reynolds, W. N., [illegible]

Type 304 stainless steel changed only slightly in room-temperature tensile properties and remained ductile after an exposure of 3 x 10^{21} n/cm^2 at 700°F. Of more interest are the properties at reactor operating temperatures; these results will be determined at Battelle Northwest Laboratories, and the results will be made generally available.

As expected, Berylco-25 and tantalum continue to show poor corrosion resistance in sodium. However, all Berylco-25 components have long since been removed from the reactor, and the tantalum-clad neutron sources are being replaced. Corrosion of the tantalum source cladding has not produced a problem in the operation of the reactor.

Tantalum is the only material that has exhibited an appreciable reduction in density. No operational problems have resulted from this phenomenon. Samples from the sources may be useful in metallurgical studies.

The neutron-shield graphite structure continues to perform satisfactorily.

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